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TECHNICAL NOTE 3257

EFFECTS OF CHEMICALLY ACTIVE ADDITIVES ON BOUNDARY LUBRICATION OF STEEL BY SILICONES

By S. F. Murray and Robert L. Johnson

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EFFECTS OF CHEMICALLY ACTIVE ADDITIVES ON BOUNDARY LUBRICATION OF STEEL BY SILICONES

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SUMMARY

A previous investigation showed that silicones, which in themselves are poor lubricants, can be made to lubricate effectively by the addition of a solvent, such as a diester, which is believed to increase the energy of adhesion between the molecule and the surface.

In the present report another method of improving the lubricating quality of silicones was investigated, namely, that of providing chemically active additives. It has been hypothesized that silicones do not maintain oxide or other reactive coatings on metal surfaces. Conventional chemically active additives and more active compounds such as a peroxide were investigated. It was found that conventional additives were not effective, but that more active materials such as the peroxide did give effective lubrication. However, all the chemically active-type additives investigated were inferior to the solvent-type additions such as the diesters previously studied.

INTRODUCTION

During the past few years, the use of silicone fluids as lubricants for specialized applications has increased steadily. They have the best viscosity-temperature relation of any known class of fluids and a high-temperature chemical stability which is at least as good as that of any synthetic lubricant now being considered for use in aircraft turbine engines (ref. 1). They therefore merit consideration as lubricants for high-temperature applications. However, the silicones are extremely poor boundary lubricants for ferrous surfaces.

An NACA research program has been directed toward finding means of improving the boundary-lubrication characteristics of silicones for ferrous surfaces. Previously reported studies (ref. 2) have indicated that blends of silicones with 30 to 40 percent by volume of various solvents

such as diesters will provide effective lubrication for steel surfaces. This result may be associated with the effect of the solvents on the geometry of the silicone molecule. Silicone-diester blends appear to have considerable promise as possible engine lubricants.

The lubrication deficiencies of silicones themselves have been associated with failure to maintain an oxide film on steel surfaces (ref. 2), where an oxide film is generally considered essential to effective lubrication. The oxide by itself may prevent welding of metal surfaces (ref. 3) and is essential to the surface chemical reactions necessary for effective lubrication with many types of lubricating fluids and additives (refs. 4 and 5).

The role of oxygen availability for surfaces lubricated by silicones has not been experimentally established. Also, there are only scattered data on the effectiveness of chemically active lubrication additives in improving lubrication of steel surfaces by silicones. It has been considered possible that chemically active additives, such as chlorine compounds, in silicones could react with steel surfaces and thus the oxide film might be replaced with a reaction product, as, for example, an iron chloride film.

A patent (ref. 6) has recently been issued describing the use of an ester of thioglycollic acid and a chlorinated saturated aliphatic monohydric alcohol ester of a lower saturated aliphatic acid as an additive blend for use with silicone fluids. The combination of a sulfur and a chlorine containing compound has been found by previous investigation (ref. 7) to be considerably better than either compound used alone. This finding is corroborated by the beneficial results, as reported in reference 6, of the combination of sulfur and chlorine in the additive as compared with the results of using either a sulfur or a chlorine additive singly in silicone.

The object of this report is to present some data indicating the influence of available active oxygen on lubricating effectiveness of silicones for steel surfaces and also to indicate the influence of conventional types of chemically active lubrication additives on lubrication of steel by silicones. The research reported herein was conducted at the NACA Lewis laboratory. Boundary lubrication experiments were made at room temperature using a kinetic friction apparatus. Reactive additives of different chemical structure were studied in various concentrations in the silicone fluid.

WORKING HYPOTHESES

The basic structure and the geometry of the silicone molecule are discussed in a previous report (ref. 2). In this reference, the primary hypothesis advanced is that solvents can change the geometry of the silicone molecule and thus affect its lubricating ability by allowing close

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packing and maximum adhesion of the molecule to the surface. The secondary hypothesis, which is also suggested in this previous report and which will be advanced herein, was concerned with the availability of oxygen or other active atoms for the maintenance of a protective film on steel surfaces sliding in contact. This hypothesis suggests that silicones prevent available oxygen or other atoms from reacting with steel surfaces to form protective films, which could be explained by the possibly impervious (to oxygen or other atoms) nature of the adsorbed surface films formed by silicones.

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EXPERIMENTAL FLUIDS

The silicone fluid used in most cases in these tests was a linear dimethyl siloxane polymer. This fluid had a viscosity of 20 centistokes at 25° C and was used as received. An organic peroxide, a series of chlorine compounds, and two conventional lubricant additives were used as listed in table I.

APPARATUS AND PROCEDURE

Friction apparatus. - The apparatus used was previously described in reference 2 and is shown schematically in figure 1. The basic elements are the rotating mild-steel disk specimen (Rockwell A-50, $2\frac{1}{2}$ in. diam.) and the cylindrical hardened (Rockwell C-60) SAE 1095 steel rider specimen with a hemispherical (3/16 in. rad.) contact tip. The rotating specimen is driven through a belt system by an electric motor coupled with a variable-speed power transmission unit. Loading is obtained by the use of dead weights which apply a force through the pulley system shown. The friction force is measured by means of four strain gages mounted on a copper-beryllium dynamometer ring and the readings are obtained from an indicating-type potentiometer calibrated as a strain indicator. The friction coefficient μ is the ratio of friction force to applied load and is reproducible to within ± 0.02.

The specimens were finished by rotating them in a drill press and rubbing the surface with successive grades of abrasive cloth. The disks were finished with grade 1/2 polishing cloth which left uniform circumferential finishing marks with a surface roughness of approximately 30 rms as measured with a profilometer. The rider specimens were finished with grade 3/0 emery cloth. Prior to use, the specimens were cleaned by the following sequence of operations: soaking and wiping in naphtha, wiping with clean cloths saturated with an acetone-benzene solution, scrubbing with moist levigated alumina powder, rinsing with water to remove the alumina, testing for cleanliness by the ability of water to wet the surface, and removing the water by successive immersion and rinsing with distilled acetone.

For all runs with each additive, the procedure to show the effect of additive concentration was a successive series of friction runs using the same sample of silicone fluid with the various concentrations made up by supplying additional additive to this original fluid. The small amount of wear debris which accumulated in the fluid had no effect on the results. A new set of specimens was used for each run. In order to obtain each individual datum point, the apparatus was started with the surfaces in contact at the desired load; as soon as the apparatus was at the proper speed (120 ft/min) and the friction readings were stable, the data were taken. The conditions of the experiment included a constant sliding velocity of 120 feet per minute, loads from 400 to 2000 grams (110,000 to 188,000 psi initial Hertz stress), and the bulk fluid at room temperature.

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RESULTS

Preliminary wear tests, which are discussed briefly in reference 8, have shown that with this apparatus, either unstable friction values or an increase in the coefficient of friction is generally accompanied by a sharp increase in the rate of wear. During the course of these experiments, the following observations were used to evaluate lubricating effectiveness:

(1) Onset of instability and high ($\mu > 0.2$) or increasing values for the coefficient of friction

(2) Surface failure (incipient or mass) of the sliding specimens observed after the run was concluded

In figure 2 are shown photomicrographs of rider surfaces which are considered typical of those obtained with (a) effective lubrication, (b) incipient failure, and (c) mass failure. Effective boundary lubrication is defined as the condition of no visible surface damage or welding with a stable friction coefficient in the range found common to boundary lubrication.

The results of the experiments described herein are presented in figures 3 to 13 and also in table I, in which are summarized the pertinent observations on each fluid with respect to optimum concentration, friction coefficient, and surface appearance. The optimum concentration of additive is defined herein as the smallest amount which will provide effective lubrication in these tests, since the use of additives has an adverse effect on the viscosity index of the silicone and would result in increased corrosion. When friction values were unstable, the maximum and minimum points were plotted and a vertical arrow was placed between them. Another label on each curve indicates the maximum load at which effective boundary lubrication (EBL) was obtained.

SILICONE WITHOUT ADDITIVES

Friction data for the 20-centistoke silicone fluid alone are presented in figure 3. As evidenced by high friction and surface welding, the silicone did not provide effective lubrication under any conditions of this experiment. Accumulation, in the straight silicone fluid, of very fine wear debris caused the fluid to be practically opaque before the experiment was concluded.

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EFFECT OF PEROXIDES

Friction data, obtained by adding various amounts of methyl-n-amyl ketone peroxide to the silicone fluid, are presented in figure 4(a). The manufacturer's data, which specify a minimum of 15.5 percent active oxygen in this compound, were used as the basis for computing the active oxygen present. A concentration of only 1.7 percent by volume of methyl-n-amyl ketone peroxide (0.25 percent active oxygen) was sufficient to lower the coefficient of friction considerably and to decrease the amount of wear debris in the fluid, but mass surface damage continued to occur (at a load of 800 g). Raising the concentration of peroxide to 3.3 percent (0.5 percent active oxygen) brought further improvement by increasing the load at which unstable friction values occurred to a value of approximately 1400 grams. A concentration of 6.7 percent peroxide (1.0 percent active oxygen) provided adequate lubrication under all conditions used, with no surface damage or welding visible on the friction specimens to loads of 2000 grams. Photomicrographs of the wear spot on the rider and the wear tracks on the disk are shown in figure 5. Figure 6 shows the riders and disks unmagnified. The fine black wear debris characteristic of silicone alone is easily visible on the set of specimens at the left.

In order to show that this effect was due to the active oxygen present and not to the methyl-n-amyl ketone, a series of friction runs showing the effect of various concentrations of methyl-n-amyl ketone in silicone are shown in figure 4(b). It is apparent that at least 30 percent by volume of the ketone is necessary to achieve the same result that 6.7 percent of the ketone peroxide provides. In concentrations greater than 30 percent, the methyl-n-amyl ketone is quite beneficial, as discussed in detail in reference 2.

Davies (ref. 9) has shown that when mineral oil is used as a lubricant for steel sliding against steel in a closed system under reduced air pressure, there is a marked increase in the rate of wear when the air pressure is reduced below 110 millimeters of mercury. The addition of 1 percent by weight of benzoyl peroxide lowers this critical air pressure to about 1 millimeter of mercury. He concluded that the peroxide continued to repair broken oxide films at an adequate rate for protection of the surfaces when the concentration of oxygen in the chamber was no longer

adequate for this purpose. It is probable that a similar effect has taken place in these silicone experiments, with the peroxide decomposing on the surface and providing an oxide layer as a lubricating film.

EFFECT OF CHLORIDES

In the previous report of solvent effect on lubrication by silicones (ref. 2), it was found that the addition of carbon tetrachloride to the silicone oil resulted in effective lubrication where a concentration of 10 percent by volume of carbon tetrachloride in silicone was used. This was in contrast to the 30 to 40 percent by volume of other solvents required, and the difference was attributed to an "E.P." effect by this compound.

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The carbon tetrachloride concentration effect is illustrated in figure 7. These data show that 5 and 7 percent concentrations result in lubrication failure (as shown by instability of the coefficient of friction) at loads of 800 and 1200 grams, respectively. The 10 percent concentration resulted in no lubrication failure at loads to 2000 grams. Because of the beneficial results with carbon tetrachloride, chlorine was chosen as a suitable element to replace oxygen.

The first series, chosen because they differed only slightly in molecular weight and geometry and contained almost the same percentage of chlorine, shows clearly the effect of the change in reactivity of the chlorine atom. Figure 8(a) is for propionyl chloride, a very reactive acid chloride. A concentration of about 2 percent by volume was sufficient to provide effective lubrication over the entire range of loads. Figure 8(b) shows that 20 percent by volume of methallyl chloride (fairly active chlorine atom) was necessary to provide effective lubrication over the same range of loads, and n-butyl chloride, which contains a relatively inactive chlorine atom, required more than 30 percent concentration for effective lubrication over the entire range of loads (fig. 8(c)). The actual concentration was not determined, but the amount of improvement obtained with 30 percent and previous experience with the use of solvents indicated that between 35 to 40 percent would be necessary.

For an aromatic series of chloride compounds, figures 9(a) and (b) show that benzyl chloride and benzotrichloride did not differ significantly, both being required in concentrations of about 10 percent by volume for effective lubrication. In figure 9(c) the effect of the chlorine substituted in the ring, where it is stabilized by resonance and therefore is not chemically active, is shown by the fact that 35 to 40 percent of p-chloroethylbenzene is required. It is probable that this material, unlike most of the other chlorine compounds, was acting in accordance with the solvent hypothesis as discussed in detail in reference 2.

In view of the results obtained with p-chloroethylbenzene, which has the chlorine substituted in the ring, it is interesting to note (fig. 10) that a methylphenyl silicone fluid, in which the phenyl groups also have chlorine substituted in the ring, gave a slight decrease in friction but lubrication failure still occurred; the improvement is therefore not considered significant. Varying the temperature of the bulk fluid from 75° to 300° F did not change the friction results. (These data are not shown.) It is probable that under these conditions the chlorine which is substituted into the phenyl side groups attached to the silicone chain will not react with the surface unless the bulk metal surfaces can reach a temperature high enough to cause appreciable decomposition of the fluid. This decomposition can occur and was probably responsible for the effectiveness of a chlorinated methylphenyl silicone in preventing mass failure at the very high surface stresses encountered in some unreported runs with a modified SAE machine.

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In order to determine the effectiveness of benzyl chloride as an additive in fluids other than silicones, runs were made with cetane, a straight-chain saturated hydrocarbon, to obtain some basis for comparing the amount of additive needed in silicone with that required for a paraffinic hydrocarbon base stock. A sample of cetane was purified by repeated percolation through columns of silica gel and fuller's earth. The results of adding various concentrations of benzyl chloride to this fluid are shown in figure 11. The concentration of additive necessary for effective lubrication with cetane is very small when compared with the concentration of the same additive needed with the silicone.

EFFECT OF VISCOSITY

An attempt was made to determine whether the viscosity of the silicone fluid would have an effect on the concentration of additive required for effective lubrication. It was felt that the use of a higher viscosity fluid might alter the rate at which the chloride compound could diffuse through the bulk fluid and the adsorbed surface layer of silicone on the steel, which was being removed during sliding, to repair the chloride film. Silicone fluids of various viscosities (10 cs and 100 cs at 25° C) were each used with carbon tetrachloride as the additive. The results showed no significant variations in the required amount (table I).

EFFECT OF OTHER CONVENTIONAL ADDITIVES

Although fatty acids are not considered to be extreme pressure lubricating additives, the effect of stearic acid on the lubricating ability of silicone fluid was considered of interest because its behavior is influenced by oxide films. Since stearic acid is insoluble and ineffective

in the silicone fluid, a mutual solvent, methylethyl ketone, was introduced. The results, shown in figures 12(b) and (c), indicate that the stearic acid is ineffective under these conditions. While the addition of 1 percent stearic acid to the blend of 20 percent methylethyl ketone and 80 percent silicone appreciably decreased the friction coefficient, lubrication failure still occurred. For the blend of 40 percent methylethyl ketone and 60 percent silicone, the addition of 1 percent stearic acid showed no significant difference. For purposes of comparison, the effect of adding 1 percent stearic acid to cetane and to methylethyl ketone is also shown (fig. 12(a)).

A typical antiwear compound, triethyl phosphate, which has been shown to be quite effective alone or as a petroleum oil additive (ref. 10), was found to be soluble in silicone up to a concentration of about 10 percent by volume. However, no improvement in lubrication was obtained with this combination of straight silicone plus 10 percent triethyl phosphate (fig. 13(a)). A series of runs made with silicone-methylethyl ketone blends using triethyl phosphate in place of stearic acid also showed no advantages to be gained by the use of an additive of this type (figs. 13(b) and (c)).

DISCUSSION OF RESULTS

The mechanisms for the behavior of additives included in this investigation are still obscured by a lack of knowledge of the surface chemistry of silicones. However, if it is assumed that in some manner the silicone inhibits oxide or reaction film formation on ferrous surfaces, the necessity for using relatively large quantities of even highly reactive compounds to obtain effective lubrication can be explained.

There are several possible ways by which oxidation of the lubricated surfaces might be inhibited:

(1) A slow rate of reaction which prevents complete repair of the film before the slider again passes over a given point. In this regard it should be mentioned that the experiments reported herein were performed at low sliding velocities and therefore, with respect to reaction rate, were less severe than should be expected in practical lubrication applications.

(2) The silicone molecule itself is preferentially oxidized at the local "hot spots."

(3) The adsorbed surface film formed by the silicone is impervious to all but the most active atoms.

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The results show that for effective lubrication under the conditions of these experiments, highly reactive compounds must be used as additives if low concentrations of additives are desired. This leads to other difficulties, especially corrosion. On the other hand, addition of larger quantities of less reactive compounds or changes in the chemical structure of the silicone molecule (such as chlorination of side groups) necessarily invoke a viscosity index penalty and may not improve lubrication except at high temperatures.

The use of solvents (such as diesters) in silicones as previously reported in reference 2 appears to be a more practical method of obtaining effective lubrication by silicones than the use of chemically reactive additives of the types discussed herein. The data of table 29 of reference 1 and unreported experience at the Lewis laboratory indicate that lubrication additives can be used with silicone-diester blends (containing more than 30 percent diester) more effectively than with silicone alone.

One silicone-solvent blend that has indicated promise as an aircraft turbine lubricant is a silicone-diester blend that has been designated NACA SD-17 lubricant. This fluid contains one-third (by volume) of di-(2-ethylhexyl) sebacate (Rohm and Haas Plexol 201), two-thirds low phenyl content methylphenyl polysiloxane (Dow-Corning 510 fluid, 100 cs at 25° C), and 0.5 weight percent of phenothiazine (Dow Chemical N.F. purified grade).

SUMMARY OF RESULTS

The following results were observed from boundary lubrication studies conducted with silicones containing chemically active additives to improve lubrication:

1. Boundary lubrication of steel surfaces by silicones can be improved by increasing the availability of oxygen through the use of a peroxide additive. Additives that could supply necessary oxygen introduce other problems and therefore cannot be considered practical at present. The effect of the peroxide additive is substantiating evidence for a hypothesis to explain the poor lubricating ability of silicones, which suggests that silicones prevent normally available oxygen from reacting with ferrous surfaces to form the oxide films that are necessary for good boundary lubrication.

2. Conventional types of chemically reactive lubrication additives must be used in very high concentrations or be highly active compounds in order to improve boundary lubrication by silicones. Such usage would result in loss of good viscometric properties or in increased corrosivity.

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3. The use of solvents (such as diesters) in silicones appears to be a more practical method of obtaining effective lubrication by silicones than the use of chemically reactive additives.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio June 11, 1954

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TABLE I. - SUMMARY OF OBSERVATIONS ON EFFECT OF ADDITIVES

Base stock	Additive	Optimum concentration, percent	Relative activity of additive	Coefficient of friction at 2000 g for optimum concentration	Condition of lubricated surface after running
Silicone (20 cs)	None	----------	----------	0.3	Mass failure
Silicone (20 cs)	Methyl-n-amyl ketone peroxide	6.7	Very active oxygen source	0.105	Effective
Silicone (20 cs)	Methyl-n-amyl ketone	40	Inactive solvent	0.13	Effective
Carbon tetrachloride	None	----------	----------	0.170	Effective
Silicone (20 cs)	Carbon tetrachloride	10	Active	0.115	Effective
Silicone (100 cs)	Carbon tetrachloride	10	Active	0.12	Effective
Silicone (10 cs)	Carbon tetrachloride	10	Active	0.12	Effective
Silicone (20 cs)	Propionyl chloride	2	Very active	0.103	Effective
Silicone (20 cs)	Methally chloride	20	Fairly active	0.103	Effective
Silicone (20 cs)	Butyl chloride	>30	Inactive	Not determined	Mass failure
Silicone (20 cs)	Benzyl chloride	10	Active	0.102	Effective
Silicone (20 cs)	Benzotrichloride	10	Active	0.108	Effective
Silicone (20 cs)	p-Chloroethylbenzene	40	Inactive	0.120	Effective
Chlorinated methyl phenyl silicone	None	----------	----------	0.220 - 0.255	Mass failure
Cetane	Benzyl chloride	1	Active chlorine source	0.145	Effective
Methylethyl ketone	Stearic acid	1	Strong tendency for chemisorption	0.130	Effective
Cetane	Stearic acid	1	Strong tendency for chemisorption	0.112	Effective
Silicone + solvent[a]	This is a "blank" reference run			0.230 - 0.349	Mass failure
Silicone + solvent[a]	Stearic acid	1[c]	Strong tendency for chemisorption	0.120 - 0.170	Mass failure
Silicone + solvent[a]	Triethyl phosphate	5[c]	Antiwear agent	0.220 - 0.322	Mass failure
Silicone + solvent[b]	This is a "blank" reference run			0.155	Effective
Silicone + solvent[b]	Stearic acid	1[c]	Chemisorption	0.160	Effective
Silicone + solvent[b]	Triethyl phosphate	5[c]	Antiwear agent	0.170	Effective

[a]Blend of 20 percent methylethyl ketone and 80 percent silicone (20 cs).

[b]Blend of 40 percent methylethyl ketone and 60 percent silicone (20 cs).

[c]Percentage additive used is reasonable quantity based on experience with mineral oils.

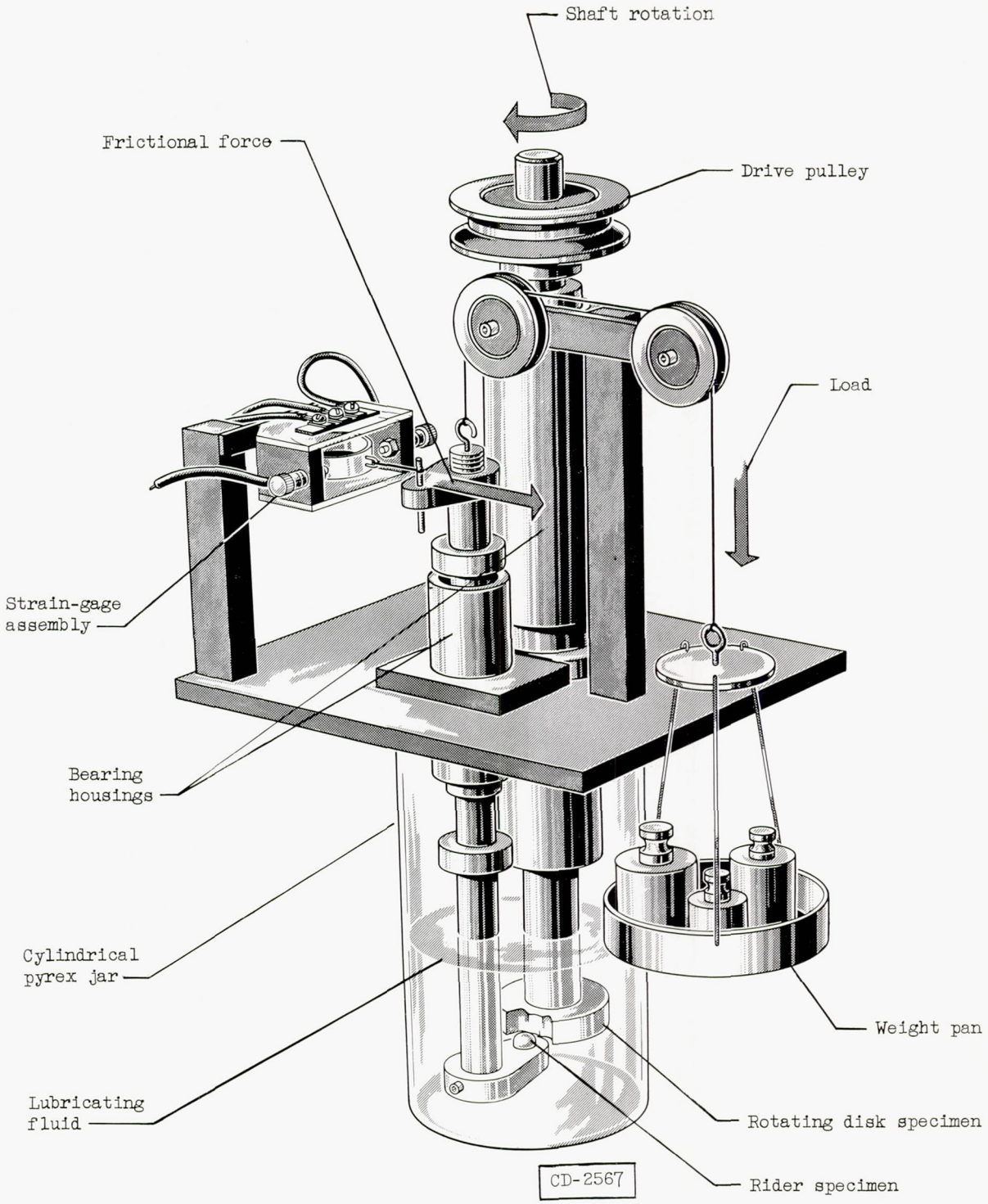

Figure 1. - Schematic diagram of friction apparatus for studying boundary lubrication by bulk lubricants.

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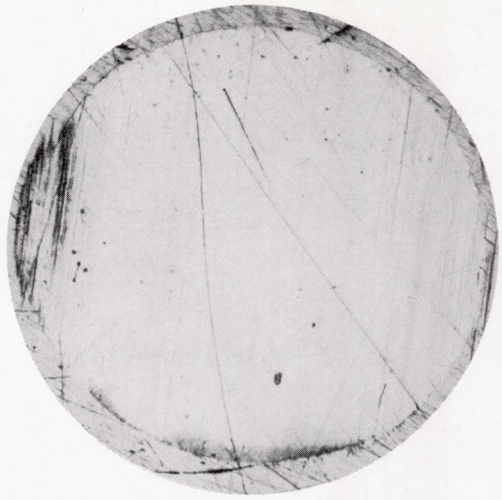
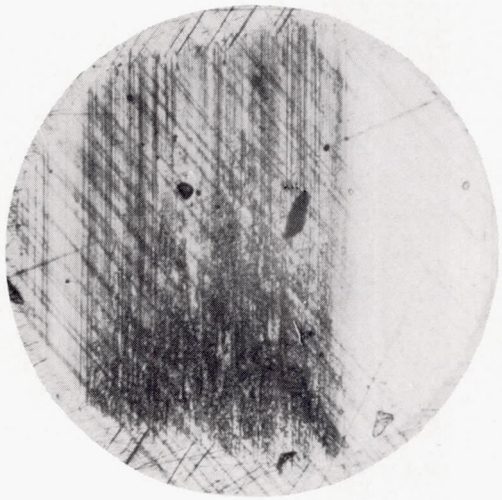
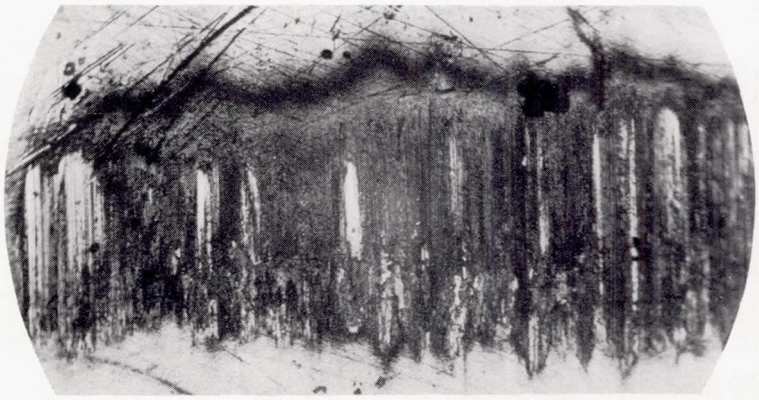


(a) Effective lubrication.



(b) Incipient surface failure.



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(c) Mass surface failure.

Figure 2. - Photomicrographs showing typical wear areas on rider specimens. X100.

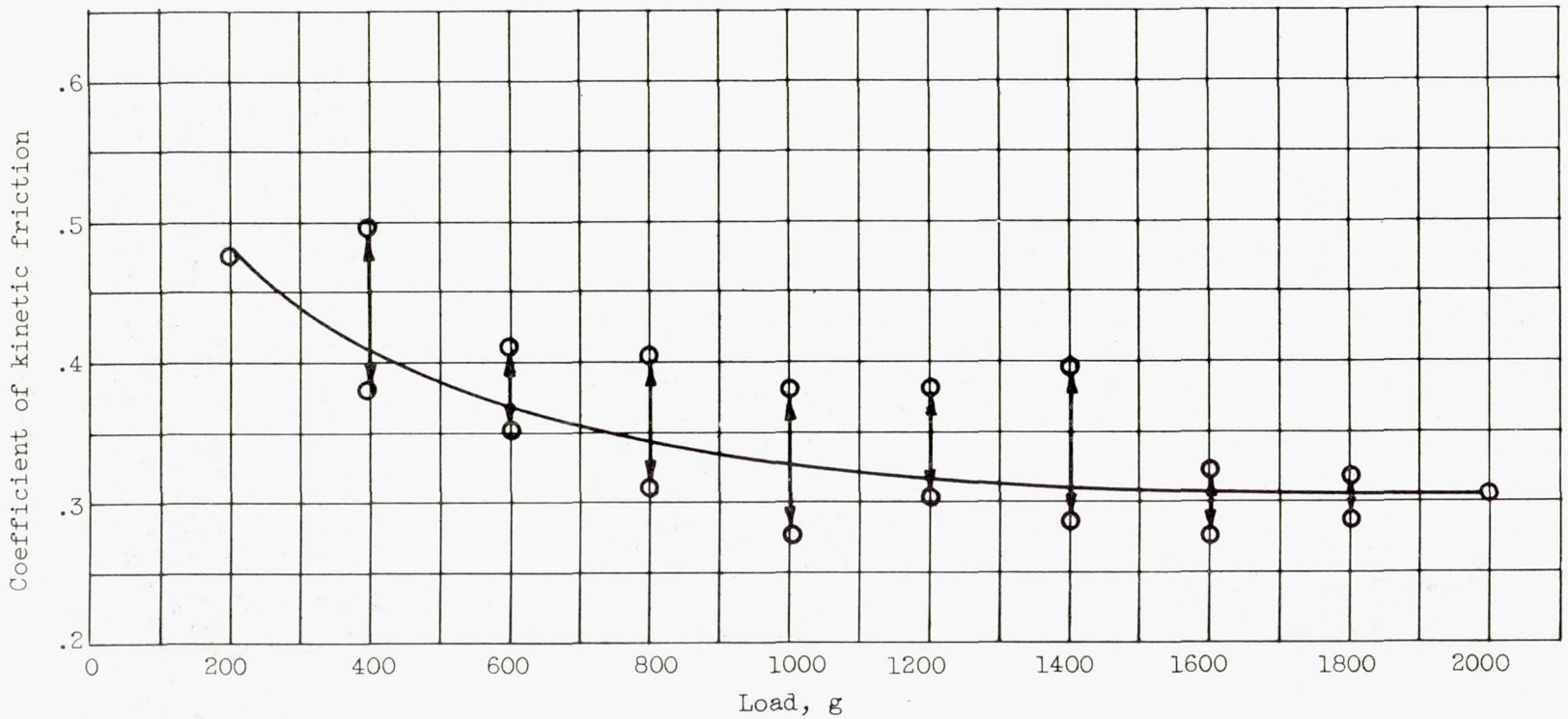

Figure 3. - Effect of load on friction of steel sliding against steel lubricated with silicone fluid. Sliding velocity, 120 feet per minute.

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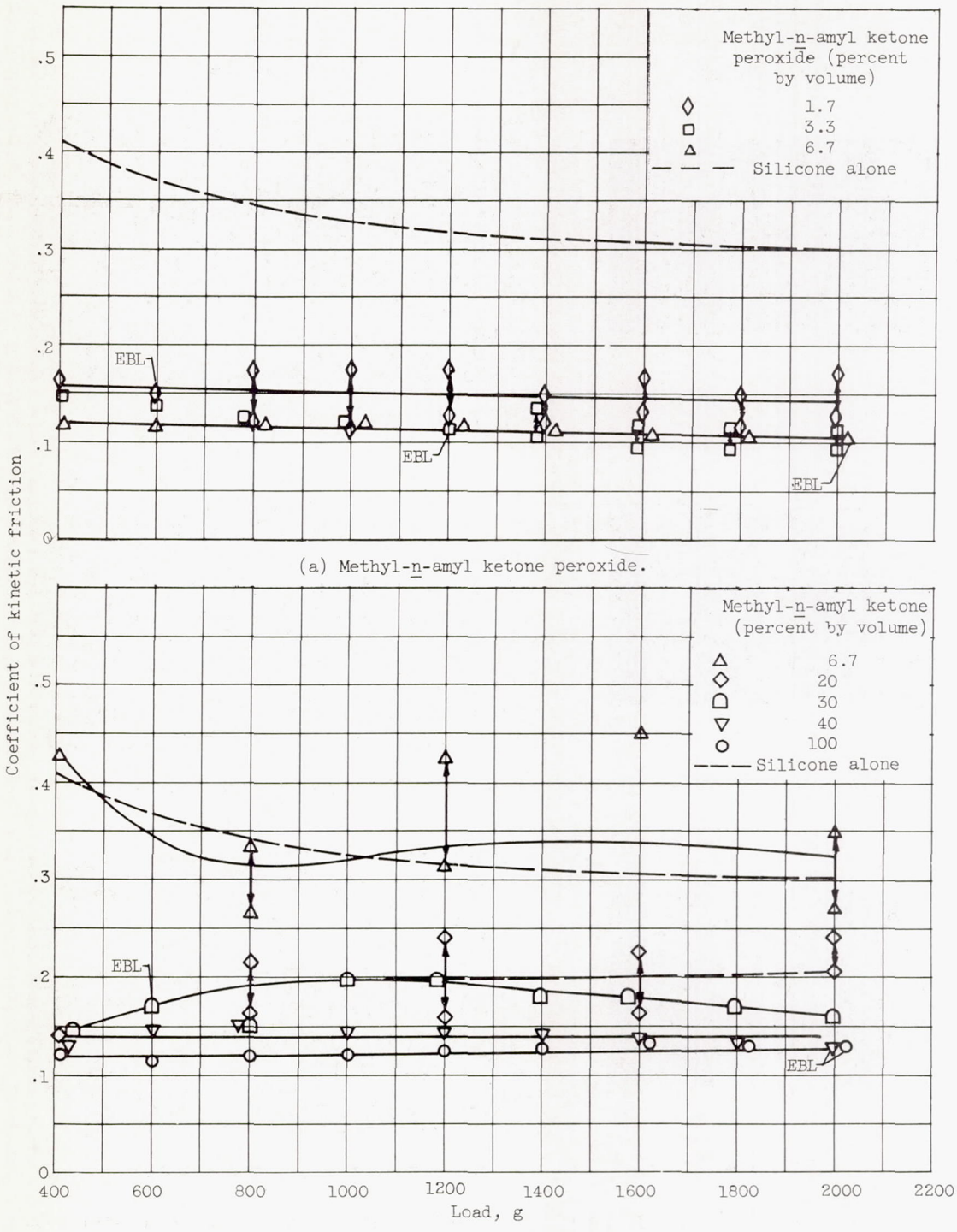

(a) Methyl-n-amyl ketone peroxide.

(b) Methyl-n-amyl ketone.

Figure 4. - Effect of concentration in silicone of methyl-n-amyl ketone peroxide and methyl-n-amyl ketone on coefficient of friction of steel against steel. Sliding velocity, 120 feet per minute; EBL, effective boundary lubrication.

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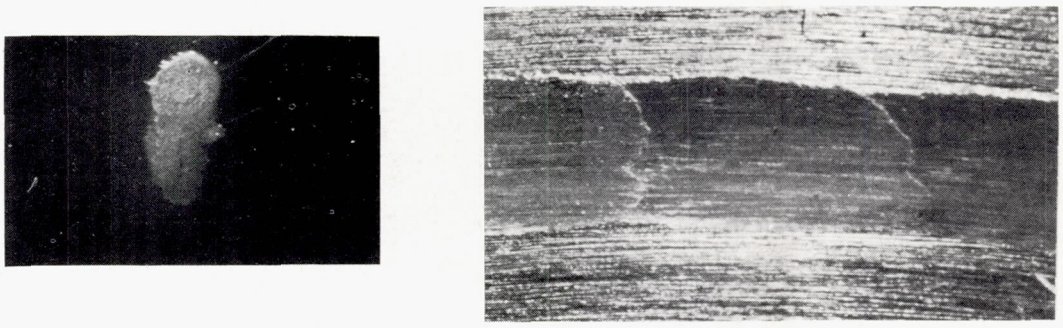
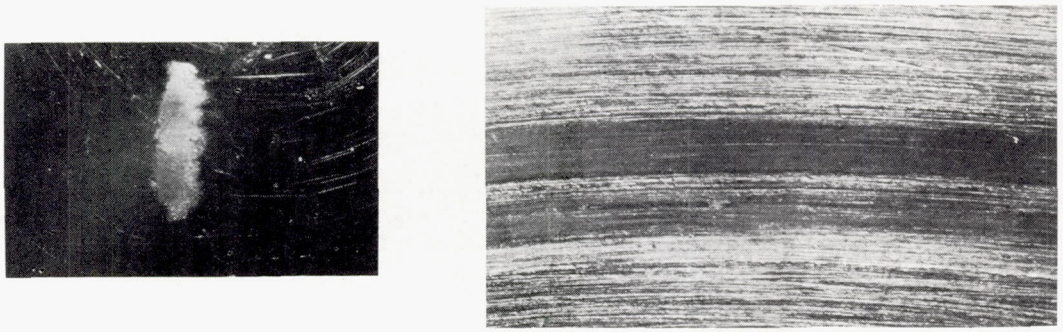
Rider specimen

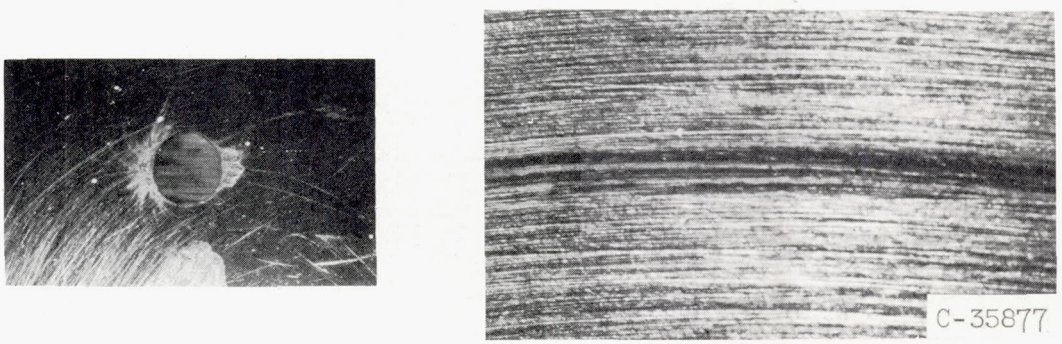
Disk specimen



(a) Silicone (20 cs).



(b) Silicone (20 cs) + 1.7 percent methyl-n-amyl ketone peroxide.



(c) Silicone (20 cs) + 6.7 percent methyl-n-amyl ketone peroxide.

Figure 5. - Photomicrographs showing effect of peroxide addition on wear areas of rider and disk specimens. X15.

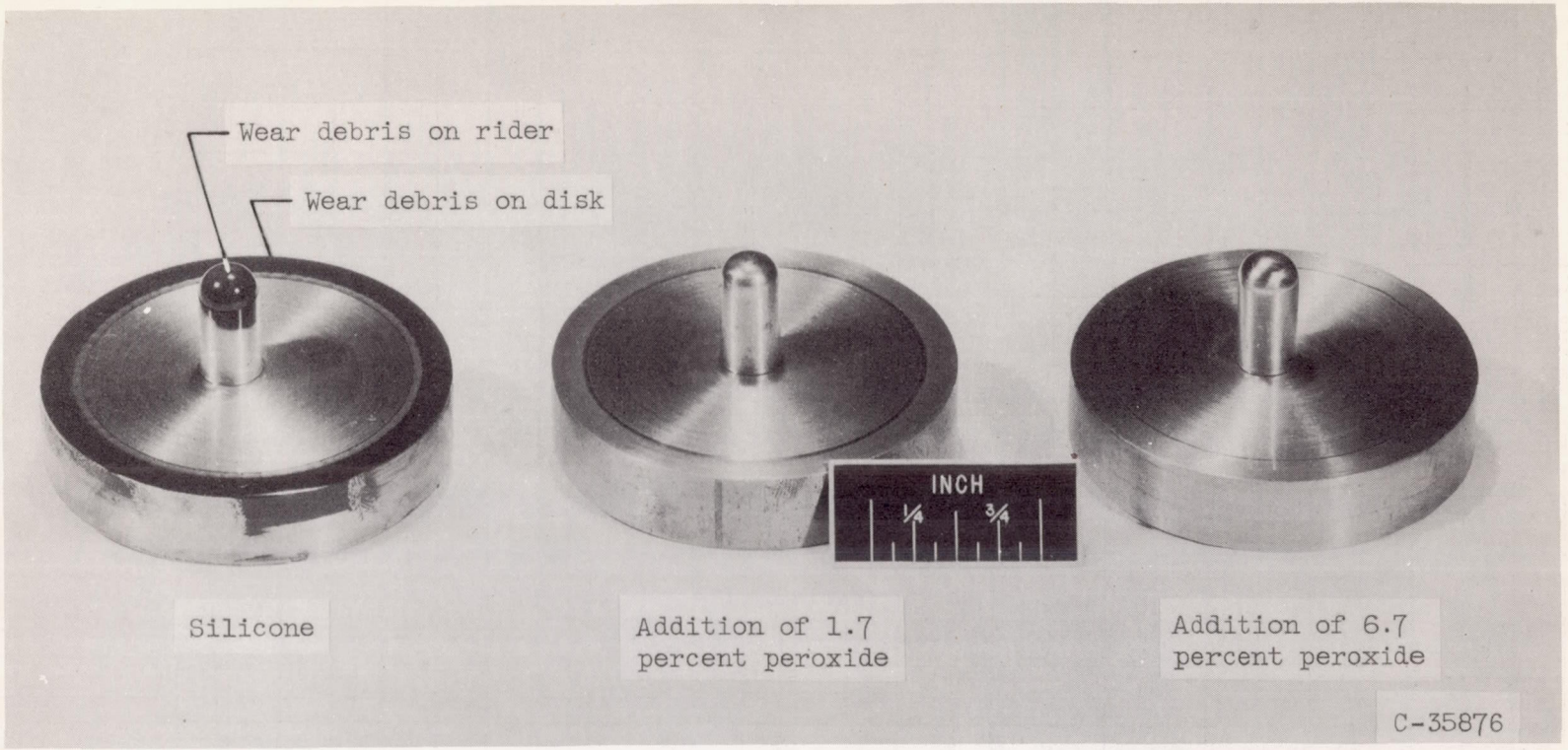
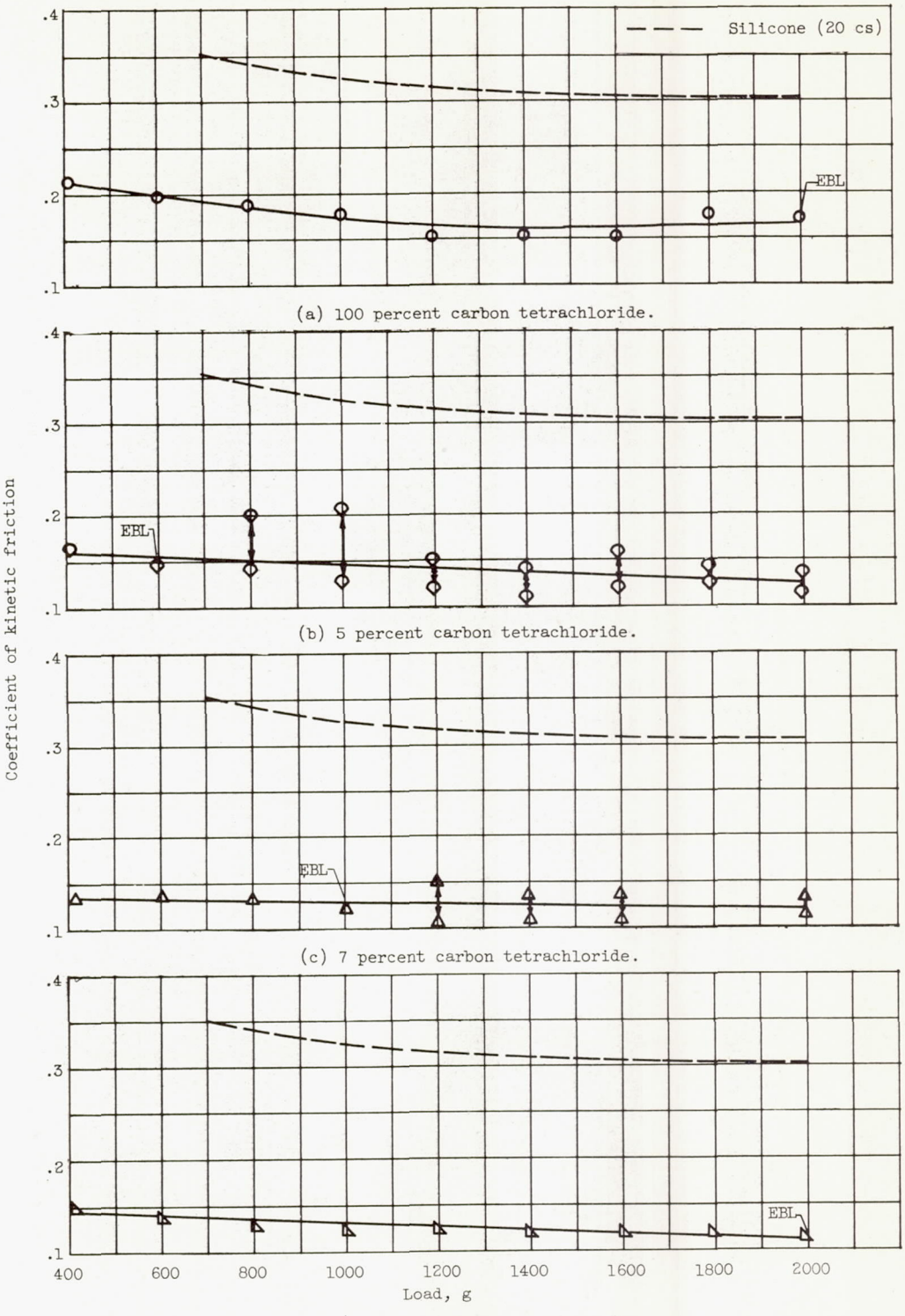


Figure 6. - Photograph of rider and disk specimens showing heavy wear debris with silicone and improvement obtained by two additions of peroxide.



(a) 100 percent carbon tetrachloride.

(b) 5 percent carbon tetrachloride.

(c) 7 percent carbon tetrachloride.

(d) 10 percent carbon tetrachloride

Figure 7. - Effect of concentration of carbon tetrachloride in silicone (20 cs at 25° C) on coefficient of friction. Sliding velocity, 120 feet per minute; EBL, effective boundary lubrication.

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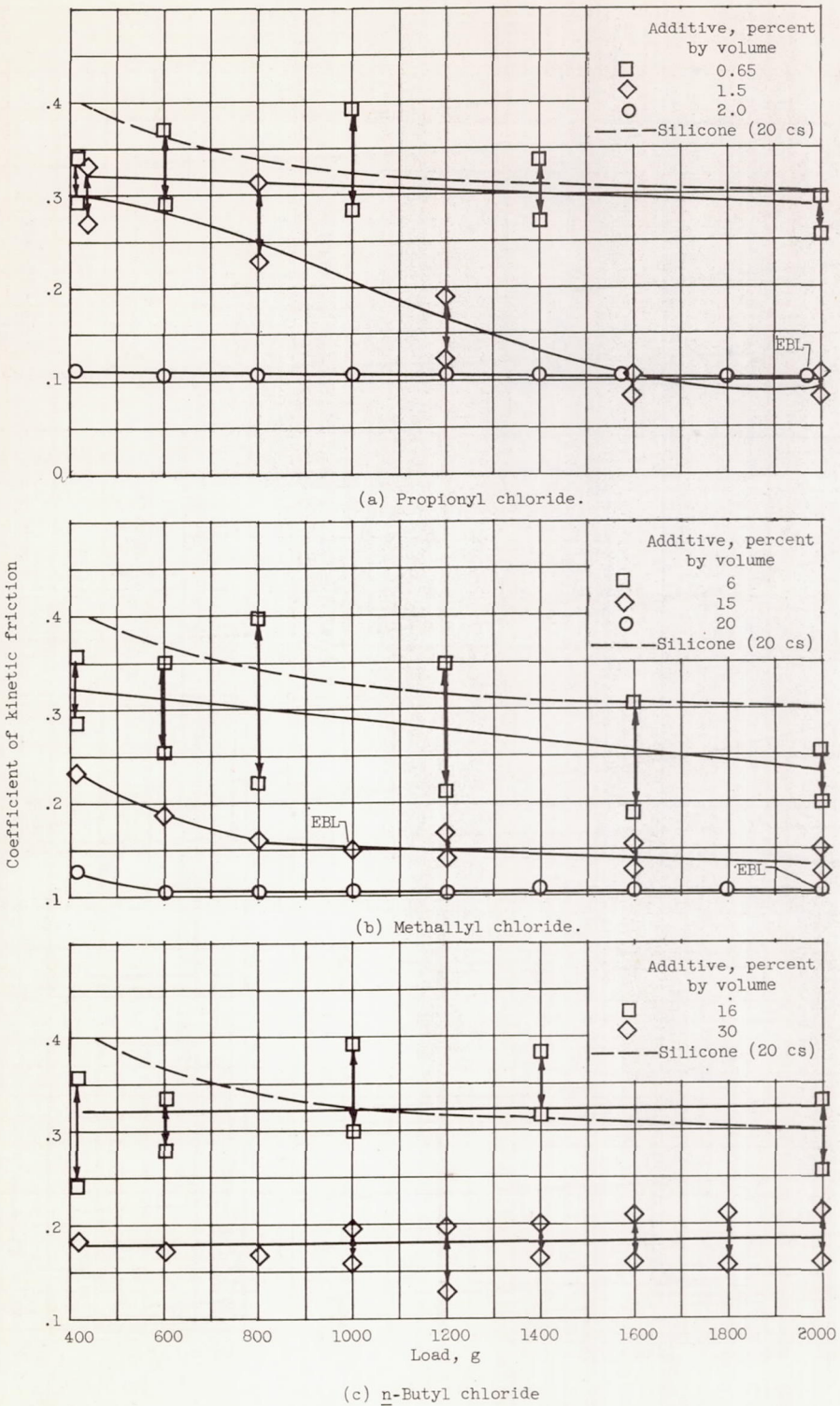

Figure 8. - Effect of concentration of a series of chlorides of different reactivity in silicone (20 cs at 25° C) on coefficient of friction. Sliding velocity, 120 feet per minute; EBL, effective boundary lubrication.

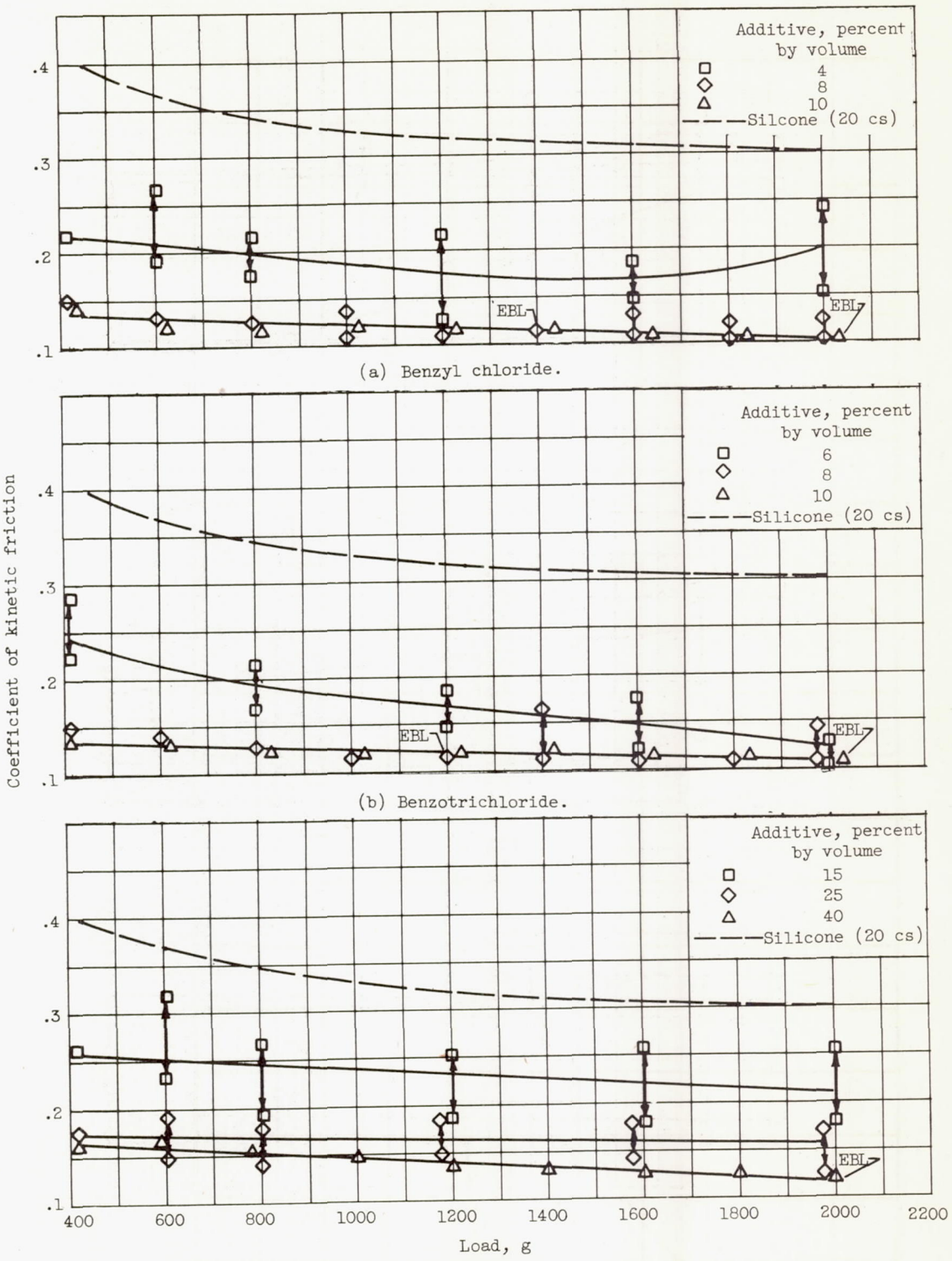

Figure 9. - Effect of concentration of a series of aromatic chlorides of different reactivities in silicone (20 cs at 25° C) on coefficient of friction. Sliding velocity, 120 feet per minute; EBL, effective boundary lubrication.

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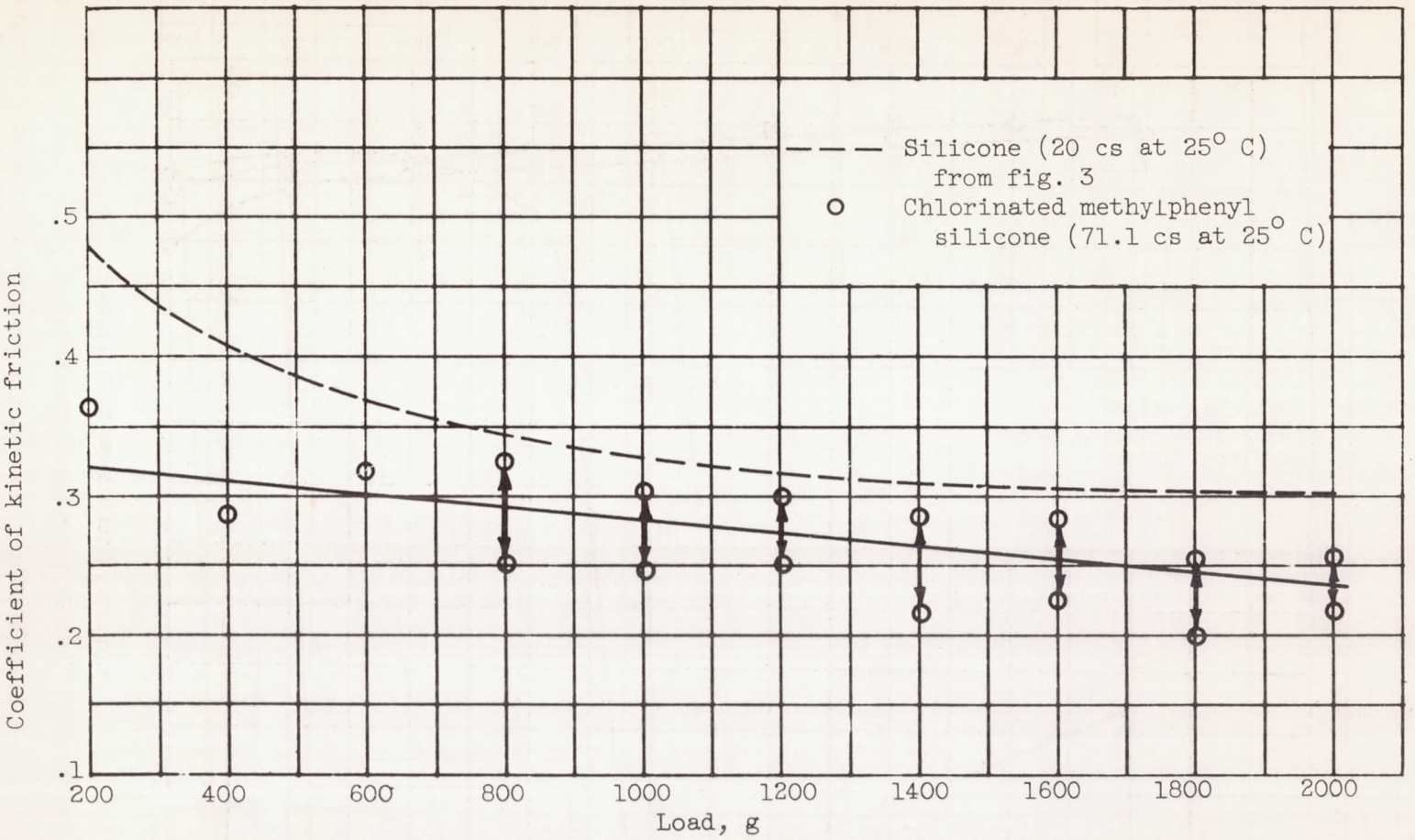

Figure 10. - Effect of load on friction of steel sliding against steel lubricated with methylphenyl silicone fluid with phenyl groups chlorinated. Sliding velocity, 120 feet per minute.

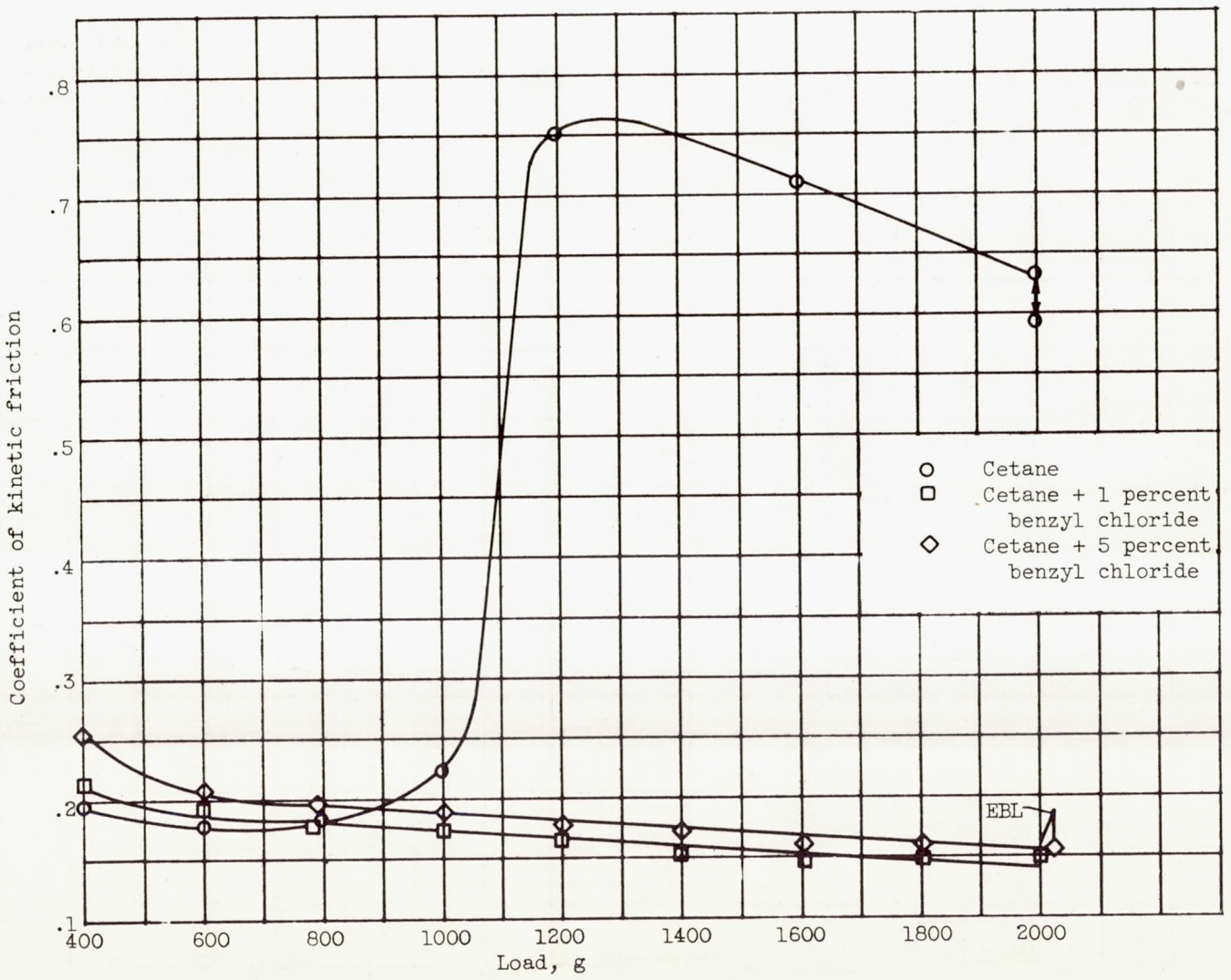

Figure 11. - Effect of concentration of benzyl chloride in cetane. EBL, effective boundary lubrication.

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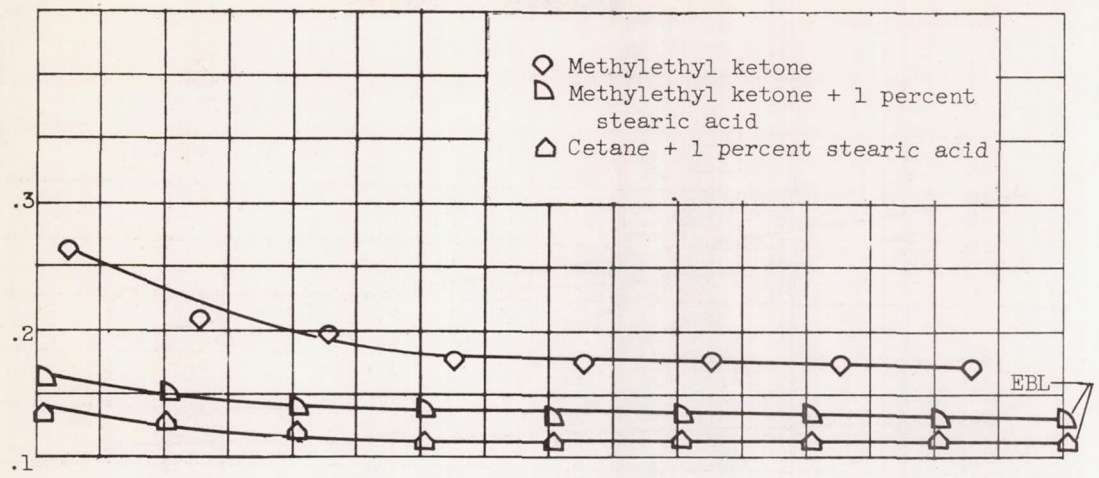
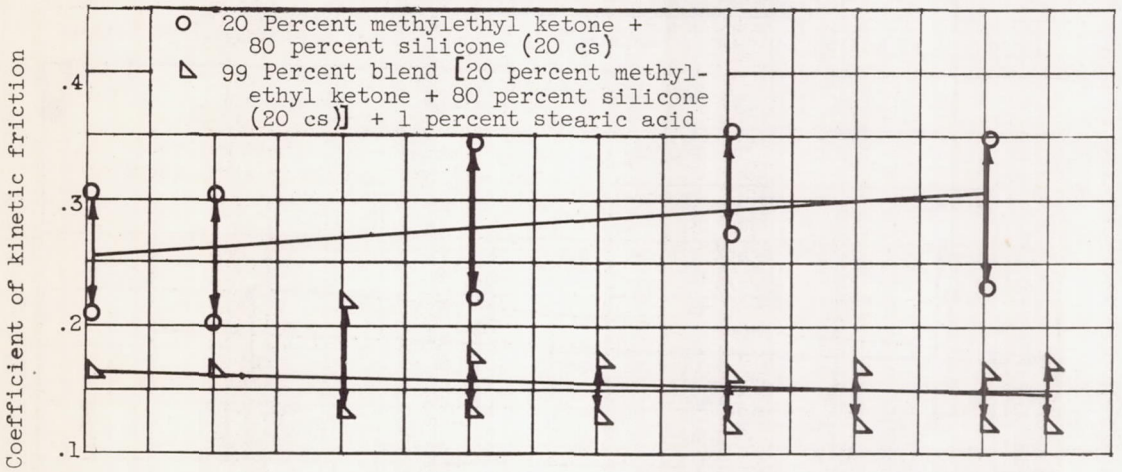
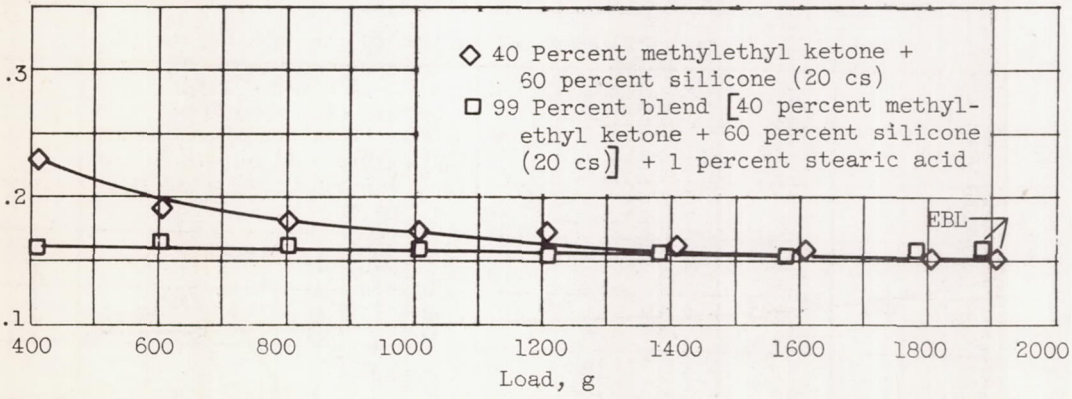


(a) Methylethyl ketone and cetane.

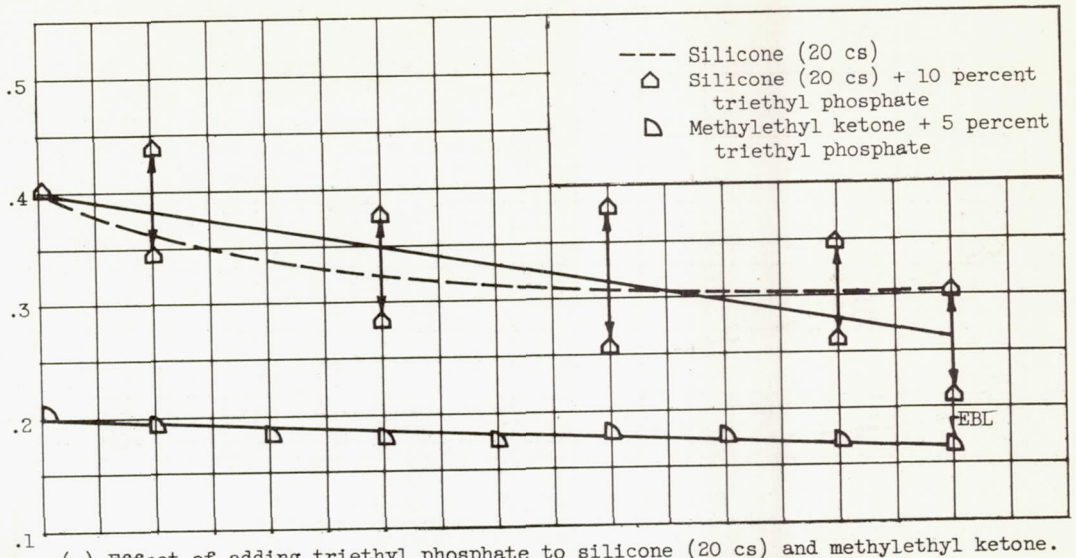


(b) Blend of 20 percent methylethyl ketone + 80 percent silicone (20 cs).

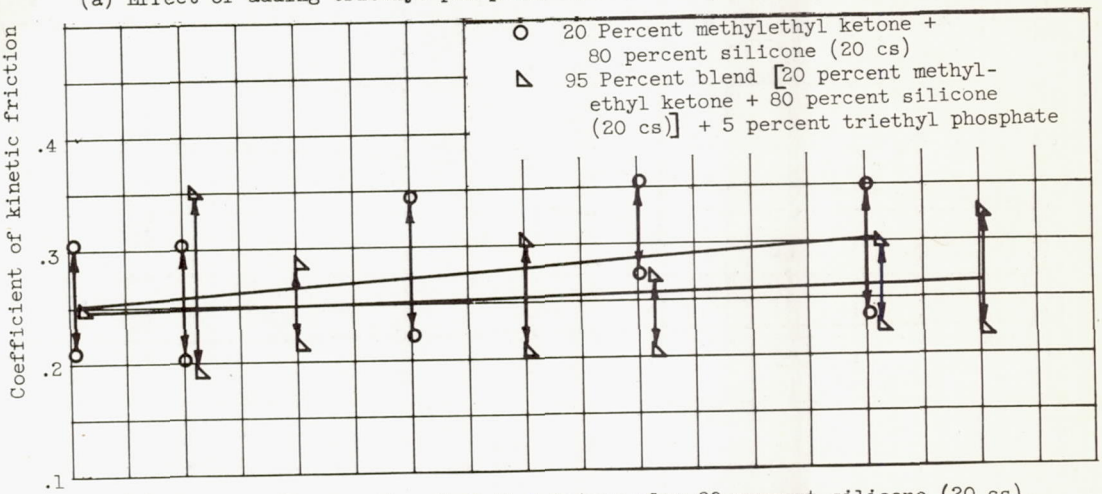


(c) Blend of 40 percent methylethyl ketone plus 60 percent silicone (20 cs).

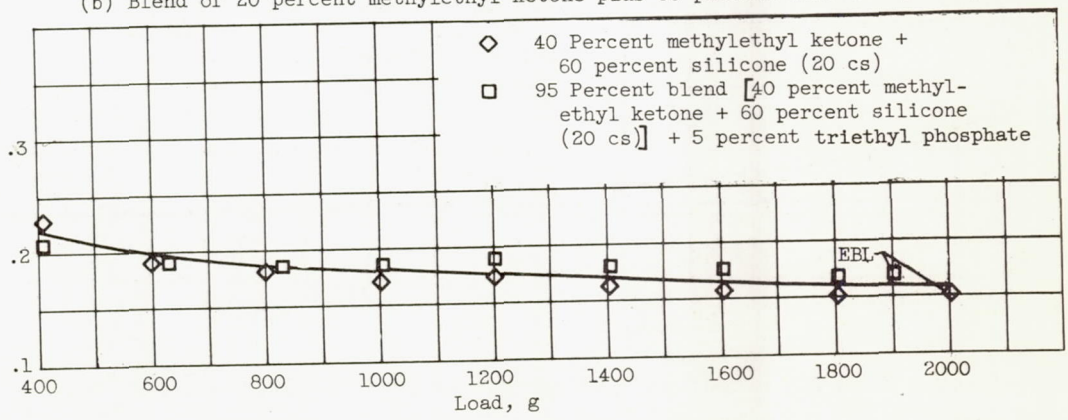
Figure 12. - Effect of adding 1 percent by weight of stearic acid to various blends of methylethyl ketone in silicone (20 cs at 25° C) on coefficient of friction. Sliding velocity, 120 feet per minute; EBL, effective boundary lubrication.



(a) Effect of adding triethyl phosphate to silicone (20 cs) and methylethyl ketone.



(b) Blend of 20 percent methylethyl ketone plus 80 percent silicone (20 cs).



(c) Blend of 40 percent methylethyl ketone plus 60 percent silicone (20 cs).

Figure 13. - Effect of adding 5 percent (by weight) of triethyl phosphate to various blends of methylethyl ketone in silicone (20 cs at 25° C) on coefficient of friction. Speed, 120 feet per minute; EBL, effective boundary lubrication.

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